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The structures of diethyl 1,14-diphenylheptacyclo[$6.6.0.0^{2,6}.0^{3,13}.0^{4,11}.0^{5,9}.0^{10,14}$]-tetradecane-2,10-dicarboxylate and of diethyl 9-oxo-2β,7β-diphenyl-1β,2,3,4β,4aα,4bβ,5α-6,7,8α,8aβ,9aα-dodecahydro-1,4:5,8-dimethanofluorene-3β,6β-dicarboxylate have been determined by single crystal X-ray structural analysis.

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11. **Structure of a Cage Dimer (I) and a Dimer Ketone (II) Formed *via* Thermal Reaction of Ethyl 3-Phenyl-2-norbornadienecarboxylate with Pentacarbonyliron**

Structure of a Cage Dimer (I) and a Dimer Ketone (II) Formed *via* Thermal Reaction of Ethyl 3-Phenyl-2-norbornadienecarboxylate with Pentacarbonyliron

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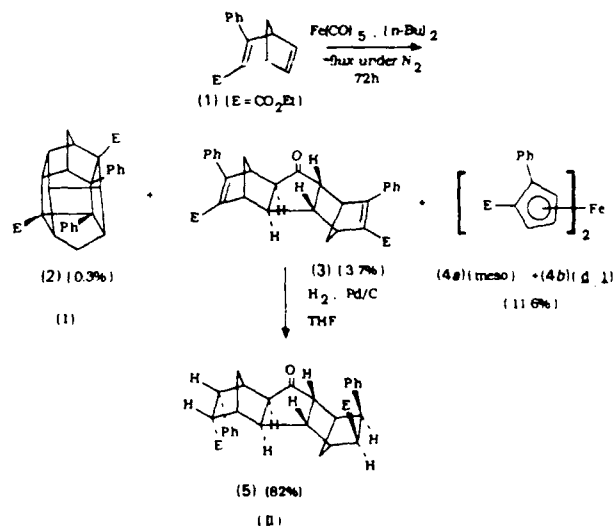
Abstract. Diethyl 1,14-diphenylheptacyclo[6.6.0.-$0^{2,6}.0^{3,13}.0^{4,11}.0^{5,9}.0^{10,14}$]tetradecane-2,10-dicarboxylate (I). $C_{32}H_{32}O_4$, $M_r = 485.60$, triclinic, $P\bar{1}$, $a = 9.292 (2)$, $b = 11.823 (2)$, $c = 13.224 (3)$ Å, $\alpha = 66.53 (2)$, $\beta = 86.80 (2)$, $\gamma = 68.36 (2)°$, $V = 1231.5 (5)$ Å^3, $Z = 2$, $D_x = 1.30$ Mg m^{-1}, λ(Mo $K\alpha$) $= 0.71073$ Å, $\mu = 0.08$ mm^{-1}, $F(000) = 512$, $T = 295$ K, final $R = 0.082$, $wR = 0.068$ for 2624 observed reflections. Diethyl 9-oxo-2β,7β-diphenyl-1β,-2,3,4β,4aβ,4bβ,5α,6,7,8α,8aβ,9aα-dodecahydro-1,4:5,8-dimethanofluorene-3β,6β-dicarboxylate (II), $C_{33}H_{36}O_5$, $M_r = 512.64$, monoclinic, $P2_1/n$, $a = 10.206 (1)$, $b = 17.853 (2)$, $c = 14.940 (2)$ Å, $\beta = 102.50 (9)°$, $V = 2657.6 (4)$ Å^3, $Z = 4$, $D_x = 1.28$ Mg m^{-3}, λ(Cu $K\alpha$) $= 1.54178$ Å, $\mu = 0.64$ mm^{-1}, $F(000) = 1096$, $T = 295$ K, final $R = 0.046$, $wR = 0.057$ for 3114 observed reflections. In (I) the cyclotetradecane cage formation occurred such that the two phenyl groups are on adjacent C atoms and all the substituents are *cis* with respect to one another (C–C–C–C torsion angles are −1.7 and −1.6° for the phenyl-ethoxycarbonyl neighbors and −26.9° for the phenyl-phenyl system). In (II) the phenyl groups are *cis* with respect to their adjacent ethoxycarbonyl neighbors (C–C–C–C torsions are −4.6 and −7.1°) but are on opposite sides of the fused ring system.

Introduction. As part of a detailed study of the stereochemistry and mechanism of the carbonyliron-promoted coupling of substituted norbornadienes to carbon monoxide (Marchand & Hayes, 1977; Marchand, Goodin, Hossain & van der Helm, 1984; Marchand, Earlywine & Heeg, 1986), the thermal reaction of Fe(CO)$_5$ with racemic ethyl 3-phenyl-2-norbornadienecarboxylate (Scheme 1) has been investigated. Reactions of this type often proceed with a high degree of regio- and stereoselectivity to afford a variety of products, among which are a heptacyclic cage cyclodimer (Neely, van der Helm, Marchand &

Hayes, 1976; Marchand & Earlywine, 1984; Marchand & Wu, 1985; Hargittai, Brunvoll, Cyvin & Marchand, 1986) and one (or more) 'dimer ketones' (Weissberger & Laszlo, 1976; Marchand & Hayes, 1977; Marchand, Goodin, Hossain & van der Helm, 1984; Marchand, Earlywine & Heeg, 1986). Indeed, such products were isolated in low yield from the corresponding reaction of (I) with Fe(CO)$_5$. A single cage cyclodimer [(2), 0.3%] and a single dimer ketone [(3), 3.7%] were isolated from this reaction along with a mixture of diastereoisomeric substituted ferrocenes [(4a) and (4b), 11.6%. Scheme 1].

Scheme 1

The structure of (2) (I) was established *via* single-crystal X-ray structural analysis. We were unable to obtain a single crystal of (3) of sufficient quality for this purpose. Instead, (3) was hydrogenated over palladized charcoal. Reduction occurred stereospecifically at the *exo* face of both double bonds in (3), thereby affording (5) (Scheme 1). A suitable single crystal of (5)

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Table 1. *Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for (I)*

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3007(5)	2887(4)	7507(3)	37(2)
C(2)	3327(5)	4038(4)	6474(3)	44(2)
C(3)	3641(5)	3532(4)	5551(3)	48(2)
C(4)	5406(5)	2681(5)	5725(4)	56(2)
C(5)	6017(5)	2726(5)	6756(4)	58(3)
C(6)	4893(5)	3975(4)	6883(4)	56(2)
C(7)	4871(5)	3537(5)	8134(4)	57(3)
C(8)	4489(5)	2326(4)	8312(3)	45(2)
C(9)	5735(5)	1587(4)	7744(4)	49(2)
C(10)	5015(5)	937(4)	7240(3)	46(2)
C(11)	5434(5)	1284(5)	6038(4)	58(2)
C(12)	3990(5)	1506(5)	5372(4)	59(3)
C(13)	2878(5)	2527(4)	5774(3)	46(2)
C(14)	3160(5)	1792(4)	7060(3)	39(2)
C(15)	1467(5)	3369(4)	7959(3)	36(2)
C(16)	1398(5)	3360(4)	9012(4)	45(2)
C(17)	-9(6)	3748(4)	9425(4)	54(3)
C(18)	-1379(6)	4158(5)	8801(5)	59(3)
C(19)	-1327(5)	4196(4)	7751(4)	54(3)
C(20)	62(5)	3810(4)	7328(4)	42(2)
C(21)	2049(5)	5401(4)	6088(4)	48(2)
O(22)	1152(4)	5914(3)	5280(3)	67(2)
O(23)	2046(4)	6008(3)	6755(3)	65(2)
C(24)	919(7)	7376(7)	6412(6)	100(4)
C(25)	-460(10)	7321(7)	6938(7)	160(6)
C(26)	5497(6)	-523(5)	7930(4)	55(3)
O(27)	6165(5)	-1098(3)	8846(3)	85(2)
O(28)	5129(4)	-1167(3)	7414(3)	73(2)
C(29)	5558(8)	-2588(6)	8031(6)	102(4)
C(30a)	4377(14)	-3034(11)	8652(16)	90(8)
C(30b)	4397(16)	-2951(14)	7718(18)	94(10)
C(31)	2196(5)	962(4)	7598(3)	39(2)
C(32)	2364(5)	274(4)	8749(4)	47(2)
C(33)	1489(6)	-481(5)	9253(4)	61(3)
C(34)	410(7)	-551(5)	8627(5)	70(3)
C(35)	219(6)	132(5)	7496(5)	67(3)
C(36)	1122(5)	867(5)	6996(4)	53(2)

Table 2. *Bond lengths (Å) and bond angles (°) for (I)*

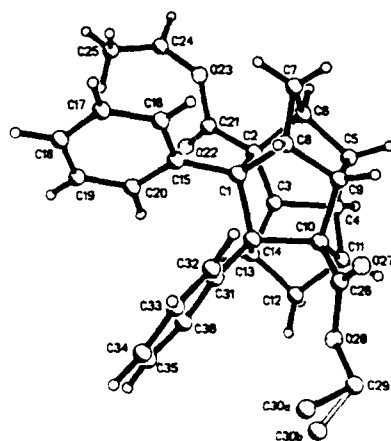|			
C(1)–C(2)	1·606(6)	C(1)–C(8)	1·548(6)
C(1)–C(14)	1·584(8)	C(1)–C(15)	1·518(6)
C(2)–C(3)	1·533(8)	C(2)–C(6)	1·546(7)
C(2)–C(21)	1·507(5)	C(3)–C(4)	1·547(6)
C(3)–C(13)	1·523(8)	C(4)–C(5)	1·534(8)
C(4)–C(11)	1·524(8)	C(5)–C(6)	1·530(7)
C(5)–C(9)	1·553(6)	C(6)–C(7)	1·524(7)
C(7)–C(8)	1·526(8)	C(8)–C(9)	1·533(6)
C(9)–C(10)	1·523(8)	C(10)–C(11)	1·541(6)
C(10)–C(14)	1·618(5)	C(11)–C(12)	1·528(8)
C(12)–C(13)	1·530(7)	C(13)–C(14)	1·555(5)
C(14)–C(31)	1·514(7)	C(15)–C(16)	1·386(7)
C(15)–C(20)	1·394(6)	C(16)–C(17)	1·377(7)
C(17)–C(18)	1·371(8)	C(18)–C(19)	1·369(9)
C(19)–C(20)	1·369(6)	C(21)–O(22)	1·190(6)
C(21)–O(23)	1·339(8)	C(24)–C(25)	1·434(9)
C(24)–O(23)	1·462(7)	C(26)–O(27)	1·196(6)
C(26)–O(28)	1·340(8)	C(26)–C(10)	1·495(6)
O(28)–C(29)	1·448(6)	C(29)–C(30a)	1·463(9)
C(29)–C(30b)	1·432(9)	C(31)–C(32)	1·396(6)
C(31)–C(36)	1·373(8)	C(32)–C(33)	1·386(8)
C(33)–C(34)	1·380(10)	C(34)–C(35)	1·372(8)
C(35)–C(36)	1·384(8)		
C(2)–C(1)–C(8)	101·1(4)	C(2)–C(1)–C(14)	104·1(3)
C(8)–C(1)–C(14)	106·6(3)	C(2)–C(1)–C(15)	113·9(3)
C(8)–C(1)–C(15)	115·9(4)	C(14)–C(1)–C(15)	113·8(4)
C(1)–C(2)–C(3)	105·1(4)	C(1)–C(2)–C(6)	102·7(3)
C(3)–C(2)–C(6)	107·3(4)	C(1)–C(2)–C(21)	115·6(4)
C(3)–C(2)–C(21)	111·4(4)	C(6)–C(2)–C(21)	113·8(5)
C(2)–C(3)–C(4)	106·0(4)	C(2)–C(3)–C(13)	108·3(4)
C(4)–C(3)–C(13)	103·9(4)	C(3)–C(4)–C(5)	104·7(4)
C(3)–C(4)–C(11)	102·6(4)	C(5)–C(4)–C(11)	108·1(3)
C(4)–C(5)–C(6)	108·3(3)	C(4)–C(5)–C(9)	104·4(5)
C(6)–C(5)–C(9)	102·6(4)	C(2)–C(6)–C(5)	100·2(5)
C(2)–C(6)–C(7)	104·7(4)	C(5)–C(6)–C(7)	104·0(3)
C(6)–C(7)–C(8)	94·8(5)	C(1)–C(8)–C(7)	104·7(3)
C(1)–C(8)–C(9)	101·8(4)	C(7)–C(8)–C(9)	103·4(4)
C(5)–C(9)–C(8)	103·5(4)	C(5)–C(9)–C(10)	106·3(4)
C(8)–C(9)–C(10)	108·3(4)	C(26)–C(10)–C(9)	111·8(4)
C(26)–C(10)–C(11)	114·2(4)	C(9)–C(10)–C(11)	107·2(4)
C(26)–C(10)–C(14)	115·2(4)	C(9)–C(10)–C(14)	105·3(4)
C(11)–C(10)–C(14)	102·1(3)	C(4)–C(11)–C(10)	100·8(4)
C(4)–C(11)–C(12)	104·4(3)	C(10)–C(11)–C(12)	105·0(4)
C(11)–C(12)–C(13)	94·4(4)	C(3)–C(13)–C(12)	103·8(4)
C(3)–C(13)–C(14)	102·0(4)	C(12)–C(13)–C(14)	104·0(3)
C(1)–C(14)–C(10)	103·4(4)	C(1)–C(14)–C(13)	106·0(3)
C(10)–C(14)–C(13)	101·1(3)	C(1)–C(14)–C(31)	114·5(4)
C(10)–C(14)–C(31)	114·0(3)	C(13)–C(14)–C(31)	116·1(4)
C(1)–C(15)–C(16)	121·9(4)	C(1)–C(15)–C(20)	120·5(4)
C(16)–C(15)–C(20)	117·6(4)	C(15)–C(16)–C(17)	121·0(5)
C(16)–C(17)–C(18)	120·6(5)	C(17)–C(18)–C(19)	118·9(5)
C(18)–C(19)–C(20)	121·2(5)	C(15)–C(20)–C(19)	120·6(5)
C(2)–C(21)–O(22)	125·3(6)	C(2)–C(21)–O(23)	112·0(4)
O(22)–C(21)–O(23)	122·7(4)	C(24)–O(23)–C(21)	116·4(4)
C(25)–C(24)–O(23)	107·6(6)	O(27)–C(26)–O(28)	121·6(5)
O(27)–C(26)–C(10)	125·3(6)	O(28)–C(26)–C(10)	113·2(4)
C(26)–O(28)–C(29)	116·4(5)	O(28)–C(29)–C(30a)	117·2(6)
O(28)–C(29)–C(30b)	107·9(7)	C(14)–C(31)–C(32)	120·1(5)
C(14)–C(31)–C(36)	122·4(4)	C(32)–C(31)–C(36)	117·5(5)
C(31)–C(32)–C(33)	120·7(5)	C(32)–C(33)–C(34)	120·5(5)
C(33)–C(34)–C(35)	119·5(6)	C(34)–C(35)–C(36)	119·7(6)
C(31)–C(36)–C(35)	122·1(5)		

Fig. 1. Diagram of (I) as determined by X-ray diffraction. The two positions for the disordered methyl groups on C(29) are shown [C(30a) and C(30b)].

(II) was obtained *via* careful fractional recrystallization from methylene chloride–hexane mixed solvent.

Experimental. (I), colorless crystal, $0.18 \times 0.20 \times 0.40$ mm, automated Nicolet R3m/V diffractometer with incident beam monochromator, 25 centered

reflections with $11 < 2\theta < 39°$ used for determining lattice parameters, $2\theta_{max} = 40°$, range of hkl: $0 \leq h \leq 11$, $-13 \leq k \leq 13$, $-15 \leq l \leq 15$. Standards 300, 030, 003, monitored every 100 reflections, with random variation of 3·0% over data collection, $\theta/2\theta$ mode, scan width $(2.0 + \Delta 2\theta_{\alpha1\alpha2})°$, scan rate a function of count rate ($8°$ min^{-1} minimum, $30°$ min^{-1} maximum), 4883 reflections measured, 4352 unique, $R_{int} = 0.012$, 2624 observed $[F_o > 3\sigma(F_o)]$. Data corrected for Lorentz and polarization effects, absorption ignored. (II), colorless crystal, $0.20 \times 0.12 \times 0.50$ mm, except where noted same experimental conditions as for (I), cell parameters from 25 centered reflections with

Table 3. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^3$) *for* (II)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	6623 (2)	3356 (1)	9956 (1)	84 (1)
O(2)	5954 (2)	4796 (1)	5912 (1)	73 (1)
O(3)	7007 (2)	5891 (1)	6247 (1)	57 (1)
O(4)	1579 (2)	3221 (1)	6376 (1)	65 (1)
O(5)	678 (2)	2155 (1)	6756 (1)	58 (1)
C(1)	4861 (2)	4856 (1)	7568 (2)	50 (1)
C(1a)	5385 (2)	4044 (1)	7648 (2)	44 (1)
C(2)	5897 (2)	5409 (1)	7348 (2)	49 (1)
C(3)	7035 (2)	5399 (1)	8248 (2)	49 (1)
C(4)	6427 (2)	4877 (1)	8876 (2)	51 (1)
C(4a)	6459 (2)	4060 (1)	8556 (2)	47 (1)
C(5)	4647 (2)	2237 (1)	8676 (2)	53 (1)
C(5a)	4623 (2)	3190 (1)	8769 (2)	46 (1)
C(6)	3215 (2)	2030 (1)	8259 (2)	50 (1)
C(7)	3017 (2)	2277 (1)	7227 (2)	49 (1)
C(8)	4288 (2)	2733 (1)	7219 (2)	51 (1)
C(8a)	4288 (2)	3470 (1)	7765 (1)	43 (1)
C(9)	5987 (2)	3512 (1)	9196 (2)	54 (1)
C(10)	4928 (2)	5056 (1)	8573 (2)	58 (1)
C(11)	5329 (3)	2257 (1)	7866 (2)	60 (1)
C(12)	6282 (2)	5310 (1)	6431 (2)	50 (1)
C(13)	7344 (3)	5907 (2)	5348 (2)	66 (1)
C(14)	8577 (3)	5465 (2)	5320 (2)	79 (1)
C(15)	1703 (2)	2622 (1)	6753 (2)	48 (1)
C(16)	-648 (3)	2389 (2)	6263 (2)	69 (1)
C(17)	-1519 (3)	2577 (2)	6909 (2)	78 (1)
C(18)	8476 (2)	5235 (1)	8192 (2)	46 (1)
C(19)	9488 (3)	5604 (1)	8789 (2)	65 (1)
C(20)	10822 (3)	5491 (2)	8773 (3)	89 (1)
C(21)	11156 (3)	5010 (2)	8147 (3)	93 (2)
C(22)	10173 (3)	4627 (2)	7556 (2)	74 (1)
C(23)	8842 (2)	4733 (1)	7581 (2)	55 (1)
C(24)	2142 (2)	2229 (1)	8778 (2)	44 (1)
C(25)	1729 (3)	1703 (1)	9343 (2)	57 (1)
C(26)	749 (3)	1869 (2)	9827 (2)	68 (1)
C(27)	147 (3)	2559 (2)	9751 (2)	67 (1)
C(28)	542 (2)	3085 (2)	9200 (2)	56 (1)
C(29)	1521 (2)	2926 (1)	8720 (2)	48 (1)

$40 < 2\theta < 55°$, $2\theta_{max} = 112°$, range of *hkl*: $0 \le h \le 11$, $0 \le k \le 19$, $-15 \le l \le 15$. Standards 400, 272, 008 showed random variation of 2·0% over data collection, scan rate ($10°$ min^{-1} minimum to $30°$ min^{-1} maximum), 4116 reflections measured, 3522 unique, R_{int} = 0·01, 3114 observed. Data reduction same as (I).

Both structures were solved by direct methods and refined by full-matrix least squares using programs provided with the Nicolet MicroVAX system (*SHELXTL*80; Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0·0003$ [0·00025 for (II)]. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). For (I), 431 parameters: coordinates and anisotropic temperature factors for non-H atoms, H atoms riding (C–H fixed at 0·96 Å) with isotropic temperature factors equal to 1·1 $\times$ U_{eq} of covalently bonded atom, one methyl group disordered with a refined ratio of ~1:1. Isotropic secondary-extinction value $p = 0·0045$ (5) applied in $F_c^* = F_c[(1·0 + 0·002(p)F_o^2/\sin2\theta)]^{0.25}$. Final $R = 0·082$, $wR = 0·068$, max. $\Delta/\sigma = 0·01$, $S = 1·76$. Final difference Fourier map values 0·32 and −0·23 e Å^{-3}. For (II), 488 parameters: same conditions as for (I). Isotropic secondary-extinction value $p = 0·0035$ (4). Final R value = 0·046,

Table 4. *Bond lengths* (Å) *and bond angles* (°) *for* (II)

O(1)–C(9)	1.211 (3)	O(2)–C(12)	1.201 (3)
O(3)–C(12)	1.337 (3)	O(3)–C(13)	1.456 (3)
O(4)–C(15)	1.202 (3)	O(5)–C(15)	1.339 (3)
O(5)–C(16)	1.455 (3)	C(1)–C(1a)	1.540 (3)
C(1)–C(2)	1.534 (3)	C(1)–C(10)	1.530 (4)
C(1a)–C(4a)	1.548 (3)	C(1a)–C(8a)	1.555 (3)
C(2)–C(3)	1.575 (3)	C(2)–C(12)	1.514 (4)
C(3)–C(4)	1.546 (3)	C(3)–C(18)	1.519 (3)
C(4)–C(4a)	1.539 (3)	C(4)–C(10)	1.532 (3)
C(4a)–C(9)	1.518 (3)	C(5)–C(5a)	1.529 (3)
C(5)–C(6)	1.559 (3)	C(5)–C(11)	1.528 (4)
C(5a)–C(8a)	1.547 (3)	C(5a)–C(9)	1.514 (3)
C(6)–C(7)	1.573 (3)	C(6)–C(24)	1.515 (4)
C(7)–C(8)	1.534 (3)	C(7)–C(15)	1.507 (3)
C(8)–C(8a)	1.547 (3)	C(8)–C(11)	1.530 (3)
C(13)–C(14)	1.493 (4)	C(16)–C(17)	1.486 (4)
C(18)–C(19)	1.377 (3)	C(18)–C(23)	1.387 (3)
C(19)–C(20)	1.382 (4)	C(20)–C(21)	1.365 (5)
C(21)–C(22)	1.368 (4)	C(22)–C(23)	1.380 (4)
C(24)–C(25)	1.388 (3)	C(24)–C(29)	1.389 (3)
C(25)–C(26)	1.387 (4)	C(26)–C(27)	1.371 (4)
C(27)–C(28)	1.365 (4)	C(28)–C(29)	1.381 (4)
C(12)–O(3)–C(13)	117.2 (2)	C(15)–O(5)–C(16)	117.5 (2)
C(1a)–C(1)–C(2)	112.2 (2)	C(1a)–C(1)–C(10)	101.7 (2)
C(2)–C(1)–C(10)	100.0 (2)	C(1)–C(1a)–C(4a)	102.7 (2)
C(1)–C(1a)–C(8a)	112.5 (2)	C(4a)–C(1a)–C(8a)	107.7 (2)
C(1)–C(2)–C(3)	103.0 (2)	C(1)–C(2)–C(12)	116.4 (2)
C(3)–C(2)–C(12)	118.9 (2)	C(2)–C(3)–C(4)	101.9 (2)
C(2)–C(3)–C(18)	120.0 (2)	C(4)–C(3)–C(18)	115.9 (2)
C(3)–C(4)–C(4a)	110.2 (2)	C(3)–C(4)–C(10)	101.6 (2)
C(4a)–C(4)–C(10)	101.2 (2)	C(1a)–C(4a)–C(4)	103.3 (2)
C(1a)–C(4a)–C(9)	106.2 (2)	C(4)–C(4a)–C(9)	112.6 (2)
C(5a)–C(5)–C(6)	110.8 (2)	C(5a)–C(5)–C(11)	100.5 (2)
C(6)–C(5)–C(11)	101.4 (2)	C(5)–C(5a)–C(8a)	103.8 (2)
C(5)–C(5a)–C(9)	112.6 (2)	C(8a)–C(5a)–C(9)	106.1 (2)
C(5)–C(6)–C(7)	101.8 (2)	C(5)–C(6)–C(24)	115.5 (2)
C(7)–C(6)–C(24)	118.8 (2)	C(6)–C(7)–C(8)	103.2 (2)
C(6)–C(7)–C(15)	119.2 (2)	C(8)–C(7)–C(15)	116.5 (2)
C(7)–C(8)–C(8a)	110.6 (2)	C(7)–C(8)–C(11)	99.9 (2)
C(8a)–C(8)–C(11)	102.6 (2)	C(1a)–C(8a)–C(5a)	107.9 (2)
C(1a)–C(8a)–C(8)	114.8 (2)	C(5a)–C(8a)–C(8)	102.2 (2)
O(1)–C(9)–C(4a)	124.1 (2)	O(1)–C(9)–C(5a)	124.4 (2)
C(4a)–C(9)–C(5a)	111.5 (2)	C(1)–C(10)–C(4)	94.1 (2)
C(5)–C(11)–C(8)	94.4 (2)	O(2)–C(12)–O(3)	123.2 (2)
O(2)–C(12)–C(2)	125.6 (2)	O(3)–C(12)–C(2)	111.1 (2)
O(3)–C(13)–C(14)	112.6 (2)	O(4)–C(15)–O(5)	123.5 (2)
O(4)–C(15)–C(7)	125.2 (2)	O(5)–C(15)–C(7)	111.1 (2)
O(5)–C(16)–C(17)	110.9 (2)	C(3)–C(18)–C(19)	118.1 (2)
C(3)–C(18)–C(23)	124.2 (2)	C(19)–C(18)–C(23)	117.7 (2)
C(18)–C(19)–C(20)	121.4 (3)	C(19)–C(20)–C(21)	119.8 (3)
C(20)–C(21)–C(22)	119.9 (3)	C(21)–C(22)–C(23)	120.2 (3)
C(18)–C(23)–C(22)	120.8 (2)	C(6)–C(24)–C(25)	120.0 (2)
C(6)–C(24)–C(29)	123.3 (2)	C(25)–C(24)–C(29)	116.7 (2)
C(24)–C(25)–C(26)	121.4 (2)	C(25)–C(26)–C(27)	120.6 (3)
C(26)–C(27)–C(28)	118.9 (3)	C(27)–C(28)–C(29)	120.9 (2)
C(24)–C(29)–C(28)	121.5 (2)		

$wR = 0·057$, max. $\Delta/\sigma = 0·01$, $S = 2·10$. Final difference map excursions 0·14 and −0·14 e Å^{-3}.*

Discussion. Atom numbering for Tables 1 and 2 follows that shown in Fig. 1; Tables 3 and 4 follow Fig. 2.† In (I), dimerization occurred involving only the norbornadiene molecules to form a heptacyclic cage having the original ring systems rotated by 90° from one another about an axis passing through the bridgehead C atoms.

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† Note that the crystallographic numbering scheme for (II) differs from that used to name the compound.
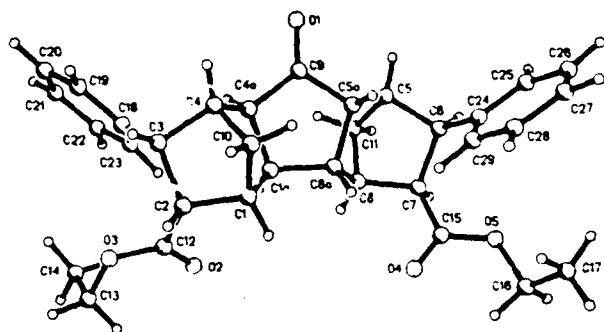
Fig. 2. Diagram of (II) as determined by X-ray diffraction.

such that two phenyl groups are bonded to adjacent C atoms. The geometry of this cage system is the same as that found in two earlier studies of norbornadiene cage dimers (Neely, van der Helm, Marchand & Hayes, 1976; Chow, Liu & Chao, 1985). The phenyl groups are cis to one another (C–C–C–C torsion is –26.9°) and also cis to their adjacent ethoxycarbonyl groups (C–C–C–C torsions are –1.7 and –1.6°). In (II), the norbornadiene dimerization occurred involving a carbon monoxide molecule to form an open, rather than caged, fused ring system. The norbornadiene moieties are trans to one another across the newly formed central planar five-membered ring. Here, as in (I), the phenyl and ethoxycarbonyl moieties on adjacent C atoms are cis to one another (C–C–C–C torsions are –4.6 and –7.1°). However, the phenyl groups are on opposite sides of the fused ring system rather than on adjacent C atoms as in (I). There are no unusually close intermolecular approaches in either molecule where

packing appears influenced solely by van der Waals forces.

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Structure of 5-Hydroxy-2,2-dimethyl-4-(p-toluenesulfonamido)-3-hexanone

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Abstract. $C_{15}H_{23}NO_4S$, $M_r = 313.41$, monoclinic, Cc, $a = 9.778$ (4), $b = 21.705$ (5), $c = 9.287$ (4) Å, $\beta = 121.07$ (3)°, $V = 1688.2$ (1) Å^3, $Z = 4$, $D_x = 1.23$ g cm^{-3}, λ(Mo Kα) = 0.71073 Å, $\mu = 1.65$ cm^{-1}, $F(000) = 672$, $T = 296$ K, $R = 0.049$ for 1028 observed reflections. The S atom has a distorted tetrahedral coordination. A bifurcated hydrogen bond is observed between the amino N atom and the carbonyl O